Product Sampling Procedure



Hazardous Waste and Toxics Reduction Program

April 2014

Product Selection and Documentation

- 1. Select products for potential analysis by consulting the QAPP¹ for the project. Select products that:
 - Are regulated by law and enforceable, depending on project goals and objectives.
 - Are likely to contain the project-targeted chemicals of concern.
 - Do not exceed a value of \$300 (according to Ecology inventory policies).
 - **Tip:** To identify where the item was purchased, keep products in store bags when cataloging them.
- 2. Photograph the shelf space around the product to document how the product is marketed if there is any ambiguity about whether the product is intended for children. Store photos according to Ecology's photo storage policies.
- 3. Save and log the receipt from the purchase. When you return to the office, scan the receipt. Save the file with the naming convention "yyyy-mm-day-storename" in the RTT > Product Testing > Store Receipts SharePoint folder. Give the original paper receipt to the Fiscal Office for credit card expense tracking purposes.

Product Tracking

- 1. Log in the product
 - Assign an identification number to the product in the "Master Product Testing Data information" spreadsheet in the RTT>Product Testing> file.
 - The ID# should be of the format "XX###-a00."
 - The XX in the code represent a two-letter designation given to each retail outlet. The codes are listed in the master product spreadsheet under the Store Codes tab.
 - The next three digits (###), start at 000, for the first product from the store and goes up by one for each product (001, 002, 003 etc.).
 - The ID number will reference all future actions to which the sample is subjected. Place the ID number on the sample bag using a label. If the product is too large for a zip-lock bag label, use a cotton string tag or place label on box.
- 2. Photograph the product before dismantling begins. Change the file name to the product identification number and upload the photo to the appropriate Product Testing > Product Pictures SharePoint folder.
- 3. Use the Product Tracking Log (Product Testing Data) to record as much of the following product information as possible:

¹ Quality Assurance Project Plan

- Product ID #.
- Product component use. Note: only needed with children-related products:
 - Intended to be put in child's mouth (e.g. teethers, pacifiers, etc.).
 - Mouthable (able to be brought to the mouth and kept in the mouth by a child so that it is sucked and chewed. If a product or part of a product in one dimension is smaller than five centimeters, it is considered mouthable.)
 - Intended to be applied to child's body (e.g., lotions, shampoos, perfumes).
 - Intended to be in prolonged (more than 1 hour) direct contact with child's skin (e.g., clothes, jewelry, bedding).
 - Intended for short (less than 1 hour) periods of direct contact with child's skin (includes many toys).
 - No direct contact with mouth or skin during reasonably foreseeable use and abuse.
- Product Category- Note: only needed with children-related products.
- Product category or brick, if known (see Ecology's Children's Safe Product Act (CSPA) reporting rule guidance on product categories).
- Age recommendation (if identified on material).
- Primary QAPP associated with project.
- For past flame retardant studies: *Harpo* + indicates that the product has a TB117 label, *Harpo* indicates that there is no label.
- Date of analysis.
- Retailer.
- Brand name.
- Associated websites.
- Manufacturer and address.
- Distributor and address.
- Importer and address.
- Date of manufacture.
- Manufacture location.
- Item/component description.

Packaging

- 1. Remove the packaging and packaging components from the product. The definition of 'packaging' and 'packaging component' is very broad and includes everything except the actual product itself. This includes all of the following:
 - Any container providing a means of marketing, protecting, or handling a product and shall include a unit package, an intermediate package, and a shipping container.
 - Unsealed receptacles, such as carrying cases, crates, cups, pails, rigid foil, and other trays.
 - Wrappers and wrapping films.
 - Bags.

- Tubs.
- Interior or exterior blocking, bracing, cushioning, and waterproofing.
- Exterior strapping.
- Coatings.
- Closures or ties.
- Inks.
- Labels.
- Hangers or other means of displaying or storing a product.
- Zippers and grommets.
- 2. Place the packaging and packaging components into a plastic bag if possible and label the bag with the Product Identification Number (PIN). If not possible, place as many items into a bag as possible and, for those portions too large, attach a label with the PIN. If you need to separate the packaging between what can be contained in a plastic bag and what cannot, make sure to attach the same PIN to both portions. For the large pieces, it may be necessary to use a string label with the PIN written on the paper portion.
- 3. Store the packaging with the Toxics in Packaging Enforcement Coordinator.

Sample Preparation

- 1. Make sure you have access to all of the necessary items on the supplies checklist in Appendix A.
- 2. Clean all needed tools as described in Appendix B.
 - Plastic tools are not recommended for samples to be analyzed for phthalates as cross contamination is possible. Plastic tools are fine for analyses of metals and parabens, as these chemicals are unlikely to be present in plastic.
 - Use of stainless steel tools are not an issue for metals analysis because the main components of stainless steel are not among the metals being tested.
- 3. Place aluminum foil on a flat surface. Place a Chem-wipe on top of the foil and replace Chem-wipes after each sample. Foil should be replaced as necessary.
- 4. If needed, perform an XRF screen on any components that could not be screened before the product was separated into components (see the "XRF Analysis" section).
- 5. Isolate product components. Refer to Appendix C for details on specific product procedures. Separate the product into individual components using cleaned tools. A product may contain multiple components in addition to any portion of the product that meets the definition of packaging.
 - Components are defined as any part of the product consisting of a different material, color, or function.

If a product is separated into several components, differentiate each component by adding an appropriate letter to the PIN and incrementing the number on the end of the PIN. Record in the Sample Log (Product Testing Data) a description of each individual component.

Each product component needs to be evaluated separately and placed into individual containers for potential analysis.

- For example, a tube of lip gloss can be separated into the product (lip gloss itself) and the container used to hold and apply the lip gloss (the external plastic tube). In this example, a scraper would be necessary to remove the lip gloss product from the plastic tube.
- Another possible product would be a compact for applying makeup. The compact is likely to
 contain powder, a puffy applicator, a mirror and the plastic container to hold all of the other
 components. Each item would be separated into potentially individual parts or components of the
 product.
- 6. Conduct XRF screen on individual components (see the following section on XRF Analysis.) This step may not be necessary depending on project goals (e.g., on lotions or perfumes being sent to the laboratory for analysis of parabens or phthalates).
- 7. Select samples for laboratory analysis. This is determined by the QAPP. If using XRF screenings for determination of sample selection, follow XRF screening levels described in the QAPP. If more samples exceed screening levels than the budget allows for, select the highest measurements across a range of metals, depending on project objectives.
- 8. Label each sample.
 - Label each component with the PIN used in the XRF scan results.
 - Place items in glass sample containers. Items may need to be reduced in size to fit inside containers. Cleaned stainless steel tools (e.g. scissors, snips, knives) should be used for size reduction.
- 9. Sample storage.
 - At the end of each day of sampling place samples in a cooler.
 - Fill out a red COC sticker and seal cooler.
 - Place cooler in COC walk-in cooler located in Ecology's headquarters basement for safe overnight storage.

10. To ship samples for lab analysis:

- Fill out a COC form. Be sure to complete all necessary fields.
- For samples to be sent to a contract laboratory, use the "COC" form from their website or provided with sample containers.
- Contact the laboratory before sending samples.

- For samples sent to Manchester Environmental Laboratory, use the "Laboratory Analyses Required" (LAR) form:
 - Contact the lab to receive the Manchester Lab work order number. Label the samples and fill out the LAR form prior to shipping.
 - The LAR form functions as a COC form.
- Protect the samples during shipping using bubble wrap.
- Place ice packs in cooler with samples.
- Fill voids in cooler with extra packing material.
- Place COC forms in a plastic bag inside cooler if sending to contract laboratory (see sample shipment below).
- Fill out and apply the security seals.
- 11. Sample shipment: Samples may be analyzed either by Manchester Laboratory or a contract laboratory and shipment to either lab follows a different procedure. For whichever laboratory is being used, follow the following practice in preparing the samples for shipment:

• Manchester Laboratory:

- Place the samples in the COC walk-in cooler either in a cage or a clearly marked cooler if there are too many samples to fit in a cage.
- Seal the cage with a numbered key or the cooler using security tape.
- Place a completed chain-of-custody (LAR) form in the courier box on the lab bench. The location of the samples must be clearly marked on the LAR. If the samples are placed in cage, identify which cage is being used and the number of the security key. The key number must be clearly identified on the LAR.
- The samples will be picked up by Manchester Laboratory courier staff and delivered to the laboratory for analysis.

• Contract laboratory:

- Add several blue ice containers to the well-packed cooler.
- Ensure that the completed COC form is in the cooler before sealing.
- Seal the cooler with shipping tape to prevent accidental release of samples during shipment.
- Add the laboratory shipping address. Take the samples to the Ecology shipping office. You will need to provide your name, phone number, and the project SIC code.
- If the samples are not going to be shipped within a short period of time, place the samples back in the sample storage refrigerator until just before shipping.
- Ship the samples using over-night services to minimize the time in transit.

XRF Analysis

1. Review XRF Standard Operating Procedure: Review the manufacturer's standard operating procedures as defined in the XL3 Analyzer Version 8.0.0 User's Guide (Abridged) Revision A November 2011.

- 2. Set up the XRF analyzer, laptop, and bench top test stand. The analyzer must be properly mounted in the bench top test stand. Plug in the analyzer's AC adapter and connection to the computer. Turn the XRF on and open the NDTr software on the computer. Make sure that the analyzer is communicating properly with the NDTr software such that it can be operated using the laptop's mouse and keyboard.
 - a. If you have trouble connecting to the XRF verify the correct USB port is being queried.
- 3. Prepare the XRF analyzer for use. The logon password is 1234.
- 4. Perform a system check.
 - a. Select "system check" on the main menu. Note: System check as the first data point saved and retrieved maintains a consistent output when downloading data from the analyzer.
- 5. Allow the XRF analyzer to warm-up: A ten-minute warm-up is advised in the manual before taking readings.
- 6. Verify performance with the manufacturer-supplied calibration check standards.
 - a. Settings:
 - i. For Flame Retardant in General Consumer and Children's project samples go to Sample Type > Consumer Goods > Plastic
 - ii. For all other analyses, go to Sample Type > Consumer Goods > TestAll
 - b. Select data entry.
 - c. Turn the camera on by clicking on the '>>' under the help tab.
 - d. Click on the typewriter image to the right of Sample to enter the run name. Name the measurement "plasticstd ##" or "metalstd ##". Conduct a two-minute scan. Repeat this standard analysis at the beginning of every shift and at the end of a session of readings. Consult the QAPP for number of times to perform standard readings between the start and end of a sampling session. Results should be within 20% of the provided values, and are usually within 10%. The standard acceptance ranges of +/- 20% are listed below:

Plastic Standard (reference sample for Niton PM 180-619, EN 71-3, lot# T-51)

Metal	Concentration	Acceptance criteria (ppm)
Cd	300	240-360
Cr	104	83-125
Pb	153	122-184
As	53	42-64
Hg	104	83-125
Ва	690	552-828
Sb	100	80-120
Se	203	162-244

Metal Standard (check sample 180-606 batch H)

Metal	Concentration	Acceptance criteria (ppm)
Ag	28,000	22,400-33,600
Cu	4,900	3,920-5,880
Cd	3,500	2,800-4,200
Pb	1,300	1,040-1,560

Note: Be careful when placing samples. The screen on the XRF is thin plastic and breakable.

- 7. Prepare the product component for screening using the XRF analyzer.
 - Ideally, the product can physically fit inside the benchtop test stand.
 - If individual components of the product cannot fit into the test stand:
 - Separate the sample into its components using the procedures described in the "Sample Preparation" section.
 - Remove the analyzer from the test stand and conduct a handheld scan.
 - If screening a liquid, gel, or any other product that may leave residue:
 - Verify the plastic sample bags being used do not contain any of the target elements. Scan the
 plastic bag alone before attempting use. It is likely the bag will need to be folded several times
 to obtain sufficient thickness for analysis.
 - To verify that the bag is not hindering screening, place the metal and plastic standards within the plastic bag and scan separately using the XRF.
 - Place the sample into a small plastic bag for analysis to avoid instrument contamination.
 - Ensure that a smooth area of the component is large enough to cover the spectrometer's window and at least 2 mm thick. If the component is less than 2 mm thick it may be folded onto itself until 2 mm depth has been reached. Take care to minimize the air trapped in between folds.
- 8. Enter identifying information into the data fields in the XRF analyzer. Each scan will have its own sample name- XX###-Y##. The first portion, XX###-, has already been assigned when logging the product in. For the second portion Y## Y represents either:
 - i. c = component of product
 - ii. p = packaging
 - iii. b = both a product component and packaging
 - iv. f = flame retardants
 - v. d = duplicate sample.

If a product is separated into several components, differentiate each component by incrementing the final two numbers, Y##, for each sample.

9. Enter the product sample ID number into the "Sample" field (XX###-Y##) by selecting Data Entry and clicking on the keyboard.

- 10. Scan the component using the XRF analyzer. Perform an initial screen of two minutes (time is automatically set for two minutes). Approximately once every 20 samples, conduct a duplicate scan (scan the same component twice (d##)).
- 11. Download the data from the XRF analyzer at the end of every session.
 - a. Close the XRF scan program and open the software used to download (NDT). Press the download icon. If system is not connected, you might need to adjust the settings and select 'connect'. Save the file in the NDT format and also check the box for "Simultaneous Download to MS Excel." Use the file naming convention "yyyy-mm-dd-XRF." Upload both files to the RTT Product Testing SharePoint folder. Download all files.
 - b. If there are problems, check settings.
 - c. Under the "Data" menu, go to Erase > Erase readings to clear data from earlier sampling event(s) from the analyzer.

To request this document in a format for the visually impaired, call the Hazardous Waste and Toxics Reduction Program at 360-407-6700. Persons with hearing loss, call 711 for Washington Relay Service. Persons with a speech disability, call 877-833-6341.

Appendix A

Supplies checklist:

Fo	r cleaning tools
	Alconox or Liquinox (cleaning agent)
	Jars to store Alconox solution
	Scrub/bottle brush
	Aluminum foil (to wrap cleaned tools)
	Chem-wipes (to dry cleaned tools)
	Metal bowl for cleaning
	Sample log (can be done on computer)
	Alcohol pads (for sterilization/cleaning)
	Absorbent pads
	Sample containers (40 mL, 2 oz, 4 oz, 8 oz, ½ L)
	Labeling stickers
	Fine tip marker
	Latex gloves
	Lab spatula
	Razor blade/carpet knife
	Scissors
	Plastic bag(s) to hold sample
	Needle nose pliers
	Standard pliers
	Various other tools that may come in handy
Sh	ipment
	Chain of Custody seals
	Chain of Custody Forms
	Sealable bubble wrap bags
	Bubble wrap/packing material
	Sample labels
То	ols
	Hammer
	Saw
	Needle nose pliers
	Pliers
	Clamps
	Drill

Appendix B

Tool cleaning:

Clean tools once at least once per day of sampling. During a sampling event (<1 day) clean the tools in between samplings using moistened alcohol wipes. Exceptions do apply. If cleaning thoroughly is entirely too impractical to be accomplished with an alcohol wipe, then a more thorough cleaning will need to be conducted as described below.

- 1. Place sufficient absorbent pads on a flat surface.
- 2. On the pads, place strips of aluminum foil that will be used to wrap the utensils used during the sample preparation. The aluminum foil strips must be large enough to encapsulate the items being cleaned. It is recommended that care be taken with the strips to prevent any potential contamination. For example, only wrap the utensils in the side of the strips that have not come in contact with the absorbent pads. You may place the strips on top of one another as long as care is taken there is no potential contamination.
- 3. Using the directions on the container, mix a liter or an appropriate amount of Alconox solution as directed on the container. Use only de-ionized water found in the sample preparation room. In addition, mix the solution in clean, unused glass sampling containers. Any unused Alconox solution can be retained for future cleanings activities.
- 4. Place an appropriate amount of the Alconox solution into a clean metal bowl.
- 5. Put on clean latex gloves.
- 6. You should only do one utensil at a time to minimize potential cross contamination.
- 7. Using a scrubber, clean the utensil with Alconox solution or Liquinox. It is recommended that the cleaning take place so that the Alconox solution does not drip back into the original bowl. Clean until all material has been removed or Alconox has come in contact with all portions of the utensil.
- 8. Using de-ionized water, rinse the utensil three times or with sufficient water to remove all the Alconox solution.
- 9. Using a clean chem-wipe, dry the utensil. Use only wipes that are directly from the box and cannot have been contaminated. Dispose of the wipe once the item has been dried.
- 10. Using a prepared strip of aluminum foil, wrap the tool in foil. Make sure that all surfaces are covered by foil and that only the internal, clean side of the foil comes in contact with the tool.
- 11. Place the cleaned tool aside to prevent contamination from work done on other utensils.
- 12. Continue with the above process until all tools are ready for use.

Appendix C

Sample Type Guidelines

The purpose of this appendix is to provide additional information on some matrices and chemical classes that might be sampled.

a. Phthalates

- i. Mainly used as plasticizers (substances added to plastics to increase their flexibility, transparency, durability, and longevity). They are used primarily to soften polyvinyl chloride (PVC) or in personal care products to carry fragrance.
- ii. Soft plastics (often clear) typical product types to target for this type of testing. An example is the clear plastic zipper bag many bed sets are sold in.
- iii. Sampling
 - 1. Plastics
 - a. Use sterilized scissors to cut into small pieces.

b. Metals

- i. Scan all metal and plastic items with the XRF.
 - 1. Packaging violations constitute any product which contains greater than 100 ppm lead, cadmium, or mercury cumulatively.
 - 2. Note: The XRF readings of cobalt are unstable; do not rely on its output in this parameter to determine testing of cobalt. Cobalt is often in blue products such as blue jeans.
- ii. Sampling
 - a. Should be reduced in size using snips.
 - 2. Small in size (e.g., zipper pull).
 - a. Send for Cryomilling.

c. Parabens

- i. A class of chemicals widely used as preservatives by cosmetic and pharmaceutical industries. These compounds, and their salts, are used primarily for their bactericidal and fungicidal properties. They can be found in shampoos, commercial moisturizers, shaving gels, personal lubricants, topical/parenteral pharmaceuticals, spray tanning solution, makeup, and toothpaste.
- ii. Parabens can be found in the ingredients lists of products.
- iii. Sampling
 - 1. Liquids
 - a. If a sample is only being tested for parabens and not volatile organic compounds, no special care needs to be taken for samples other than to minimize contamination and ensure enough volume for proper testing.
 - 2. Several small samples.
 - a. If a product contains multiple small samples, for example an eye shadow palette, make a composite sample to maximize sample volume.
- d. Volatile Organic Compounds
 - i. Generally from cosmetic or hygiene products.
 - ii. Organic samples should go directly from the original container to the sample container.
 - iii. Sampling

1. Liquids, gels, powders.

- a. If the sample is less than the volume of the container, sometimes it is best to forgo XRF measurements or only use a very small amount to measure.
- b. Once a sample has been analyzed by XRF it has been exposed to the environment too long to be an ideal organic sample. Therefore, use different bits of the sample for XRF and organic sampling.
- c. Minimal headspace is very important when sampling for the volatile organic compounds. In particular, keep this in mind when purchasing samples. Purchase duplicates if necessary.
- d. The smallest volume sample container (40 mL) is most often the best option.
- e. When ample amounts of a component are available, fill the sample container full enough that a meniscus of excess is at the top. Also, put enough sample into the lid of the container to coat the bottom surface of the cap.
- f. Carefully tilt lid and container together and screw shut. The goal is to have as little air in the sample container as possible.
- g. There should be excess sample expelling from the lid and container seam if done properly.
- h. Check for bubbles once sealed.
- i. Use a chem-wipe to clean the outside of the sample container.
 - i. Using a sticky label, indicate what sample the container is and be sure to enter it into the Sample Log (<u>Product Testing Data</u>).

e. Formaldehyde

- i. Formaldehyde releasers are often used as an antimicrobial preservative in cosmetics. Examples that might be found in ingredients lists include:
 - 1. Quaternium-15
 - 2. Imidazolidinyl urea (Germall 115)
 - 3. Diazolidinyl urea (Germall II)
 - 4. DMDM hydantoin (Glydant)
 - 5. 2-Bromo-2-nitropropane-1,3-diol (Bronopol)
 - 6. Tris(hydroxymethyl) nitromethane (Tris Nitro)
 - 7. Hydroxymethylglycinate (Suttocide A)
- ii. Formaldehyde is also a concern in wrinkle free clothing.
- iii. Sampling
 - 1. Fabric
 - a. Cut into small pieces using scissors or rotary cutter and mat.
 - 2. Liquids
 - a. See above (in Volatile Organic Compounds).

f. Flame Retardants

- i. Are found in plastics and textile applications, e.g., electronics, clothes, and furniture. Including, but not exclusively, plastic covers of television sets, carpets, paints, upholstery (internal foam), and domestic kitchen appliances.
- ii. The ½ Liter or 8 oz jars are recommended, depending on the density of the sample material to ensure there is adequate sample.
- iii. Foam samples (presumably from furniture) will need to be cryomilled. However, fabric samples should not need to be cryomilled. Plastics should be broken into pieces and milled.

iv. Sampling

- 1. Foams
 - a. Collect about 15 grams for cryomilling
- 2. Plastics
 - a. Need to be broken down into smaller pieces for cryomilling.
- 3. Fabrics
 - a. See above (in formaldehyde).